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Title

OPTICAL CONSTANTS OF METALS IN THE VACUUM ULTRAVIOLET REGION

by

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CHAPTER I

INTRODUCTION

In the past, the problem of measuring the optical constants of real materials has been attacked experimentally by measuring reflectivities and transmissivities and calculating n and k from these measurements.[1,2] These quantities, especially when obtained through a reflection technique, must perforce be very sensitive to the surface conditions of the sample to be investigated. More recently, electron eigenloss experiments, in which the incident electrons give up quantized amounts of energy in exciting collective oscillations of the conduction electrons, have become an increasingly popular approach to the optical constants problem. Unfortunately the results obtained from these two techniques do not agree well,[3] probably because of a variance in the past history of the samples used by the different experimenters.

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[1] G. L. Weissler, W. C. Walker, J.A.R. Samson, and O. P. Rustgi, J. Opt. Soc. Am. 48, 71 (1958).

[2] O. P. Rustgi, J. S. Nodvik, and G. L. Weissler, Phys. Rev. 122, 1131 (1961).

[3] L. Marton, J. Quant. Spectrosc. Radiat. Transfer 2, 671 (1962).

The theory of a quantum plasma in the volume of a metal was first developed by Bohm and Pines.[4] According to this view the interior of a metal is to be regarded as an electron gas of high mobility in a background of positive charge which is relatively fixed in space. On a time average there will be fluctuations in charge density, thermal or otherwise. As a consequence of the electron mobility there may be longitudinal oscillations of the electron gas, similar to sound waves, which are different from the classical plasma oscillations in gas plasmas in that the oscillation frequencies are so high (about 10^{16} sec^{-1}) that the associated energy, $h\nu_p$, is large compared with kT. In addition, the electron densities in metals are so great compared with those in gases that the electron gas is degenerate and its motion must be treated by Fermi-Dirac statistics rather than classical mechanics. The fact that the excitation energies of the plasma oscillations are so much larger than kT, in fact greater than the kinetic energy of any electron of the metal at ordinary temperatures, means that the oscillations will remain in their ground states unless excited by some other method such as high energy electron bombardment.[5]

If a beam of fast electrons is shot through a metal

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[4] D. Bohm and D. Pines, Phys. Rev. 82, 625 (1951).

[5] S. Raimes, Reports on Progress in Physics 20, 1 (1951).

film the electrons have a good chance of exciting one or more modes of plasma oscillation. Therefore, we should expect some of the emergent electrons to have lost energy $h\nu_p$, others $2\ h\nu_p$, and so on. Pine has given the name plasmon to such a quantum of plasma energy.[6] Many experiments have been carried out in which electron beams of several kilovolt energy have been shot through metal films of about 100Å thickness, and the energy losses determined. The plasmon energies are about 10 eV but the higher energy electron beams are easier to handle experimentally, although it is difficult to measure accurately an energy loss of 10 eV for an electron of kinetic energy of several kilovolts. In any case, for some metals the emergent electrons showed energy losses (eigenlosses) at almost exact integral multiples of the plasmon energy computed for those metals.[6] On the other hand, some non-metals also produce eigenlosses in transmitted electron beams. In some metals agreement between the theoretical plasma frequency and experimental energy loss spectra is completely lacking.

An alternative experimental approach to electronic excitation in metals was suggested by Ferrell.[7] His model of plasma oscillations in a metal predicted that, in a sufficiently thin foil, plasma oscillations should be able

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[6]D. Pines, Rev. Mod. Phys. 28, 184 (1956).

[7]R. A. Ferrell, Phys. Rev. 111, 1214 (1958).

to decay by the emission of a photon of the oscillation frequency. His estimates of the probability of such an event indicate the feasibility of an experimental approach in which the plasma oscillations in the foil are excited by electron bombardment and the emitted photons are detected spectrographically. Ferrell predicts the angular distribution of the emitted photons, their polarization, and dependence of the number of photons on foil thickness and the energy of the electron beam. The frequency of the photons should be the plasmon frequency which can be predicted from the optical properties of the material.

As suggested by Ferrell, experimental work on Ag has been done by Steinmann and others.[8,9] The early results[8,9] seem to indicate the existence of plasmons of 3.75 eV in Ag as predicted by Ferrell from optical data. Subsequent theoretical work using a model in which "transition radiation" is emitted when a point charge crosses an interface has yielded predictions[10-12] somewhat different

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[8] W. Steinmann, Phys. Rev. Letters 5, 470 (1960); Z. Physik 163, 92 (1961).

[9] R. W. Brown, P. Wessel, and E. P. Trounson, Phys. Rev. Letters 5, 472 (1960).

[10] I. M. Frank and V. I. Ginsburg, J. Phys. (USSR) 9, 353 (1945).

[11] V. P. Silin and E. P. Fetisov, Phys. Rev. Letters 7, 374 (1961).

[12] E. A. Stern, Phys. Rev. Letters 8, 7 (1962).

from those of Ferrell. Measurements on films of Ag, Al, Au, and Mg[13] have tended to fit the "transition radiation"[14] model better than the "plasmon" model. Since agreement between theory and experiment is still not good, and may be fortuitous,[13-18] work has been undertaken at this laboratory to extend the data to shorter wavelengths and to clean experimental conditions.

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[13] C. J. Powell and J. B. Swan, Phys. Rev. <u>116</u>, 81 (1959).

[14] C. J. Powell and J. B. Swan, Phys. Rev. <u>115</u>, 869 (1959).

[15] C. J. Powell and J. B. Swan, Phys. Rev. <u>118</u>, 640 (1960).

[16] R. H. Ritchie, Phys. Rev. <u>106</u>, 874 (1957).

[17] E. A. Stern and R. A. Ferrell, Phys. Rev. <u>120</u>, 130 (1960).

[18] A. L. Frank, E. T. Arakawa, and R. D. Birkhoff, Phys. Rev. <u>126</u>, 1935 (1962).

CHAPTER II

APPARATUS

The research program planned here involves emphasis on optical properties of solids, especially thin films. Since so much of the earlier data on optical properties is subject to ambiguity because of unknown surface conditions, it was felt necessary to prepare films under ultra-high vacuum and keep them under ultra-high vacuum until the measurements are completed. In this way it may also be possible to study the alkali metals which should be more amenable to theoretical calculations.

Because of the extremely high contamination rates of surfaces maintained under ordinary high vacuum conditions (about 10^{-6} Torr) the need was felt for an apparatus in which a clean metal surface could be prepared and reflectivity and other measurements made before the surface properties of the film could be altered by the residual gases of the experimental chamber. Since the monolayer forming time at 10^{-6} Torr is about one second, base pressures of the order of 10^{-9} to 10^{-10} Torr are required to achieve surfaces that remain clean long enough to perform an experiment. These pressures are in the ultra-high vacuum region and their attainment requires the special techniques associated with that field. Of primary importance is the reduction of backstreaming pump oil vapors, either by highly efficient trapping or by removal altogether of the diffusion pump. Elimination of outgassing from elastomer gaskets and acceleration of general system outgassing so that the ultimate pressure may be reached in a reasonable time are also necessary. To this end an oil diffusion pumped stainless steel ultra-high vacuum chamber has been constructed. The pumping column is shown schematically in Fig. 1 and photographically in Fig. 2. The system includes, above the 1500 liter/sec diffusion pump, a nonbakeable water cooled baffle and a refrigerated (Freon 22) baffle, two bakeable liquid nitrogen traps, and finally the ultra-high vacuum experimental volume itself. All of the above mentioned traps are optically dense and all, save the water-cooled baffle, are equipped with oil creep barriers. Wherever possible fusion welds on the inside of the vacuum envelope have been used. Great care has been taken to avoid high vapor pressure materials such as Zn, P, Cd, etc. The sealing medium in the bakeable portion of the system is OFHC Cu.

Demountable Seals. All of the demountable bakeable seals used in the system are of the pinch type copper seal.[19] This seal involves a flat OFHC copper

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[19]T. Batzer and R. Ullman, Engineering Note, Lawrence Radiation Laboratory, University of California, May 24, 1960, and October 7, 1960.

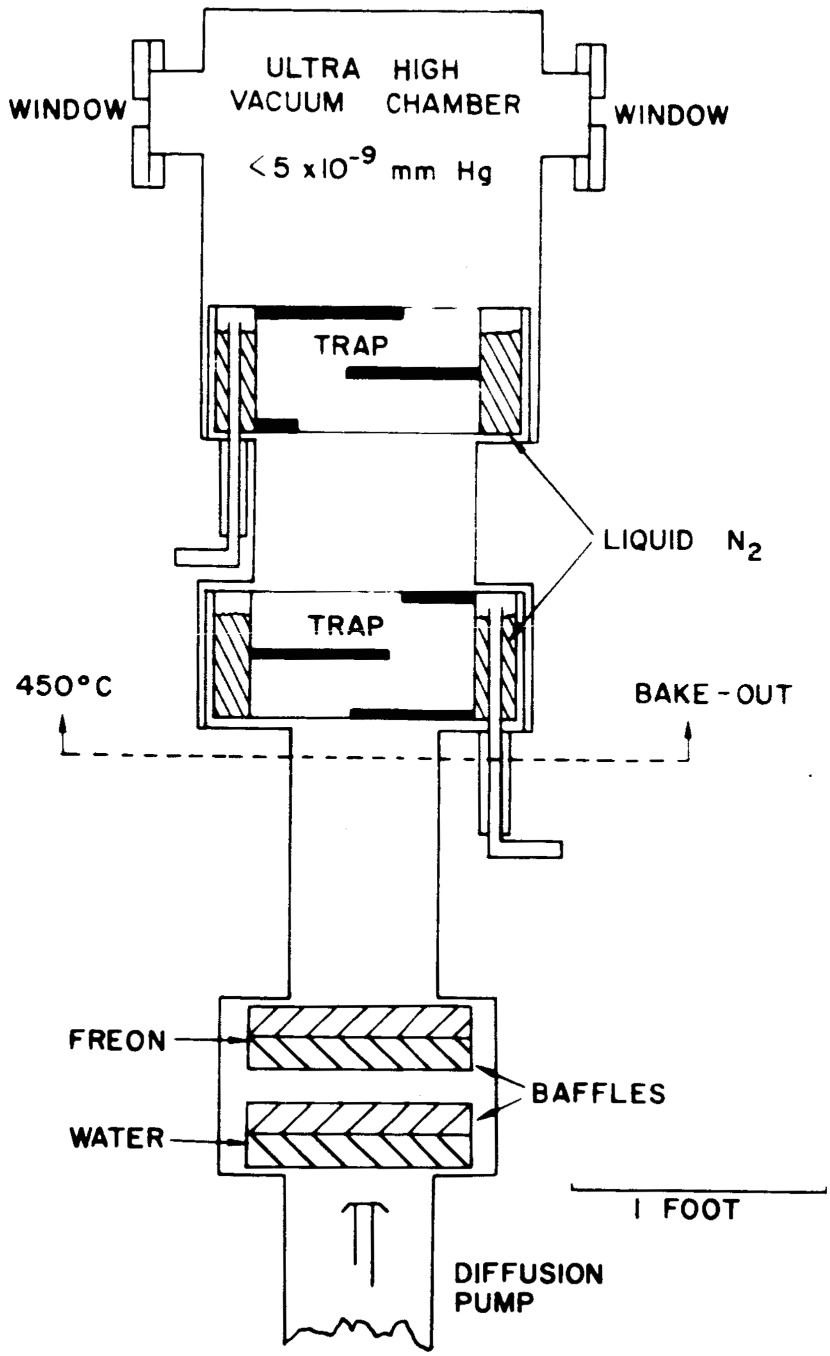

Figure 1. Ultra-High Vacuum System

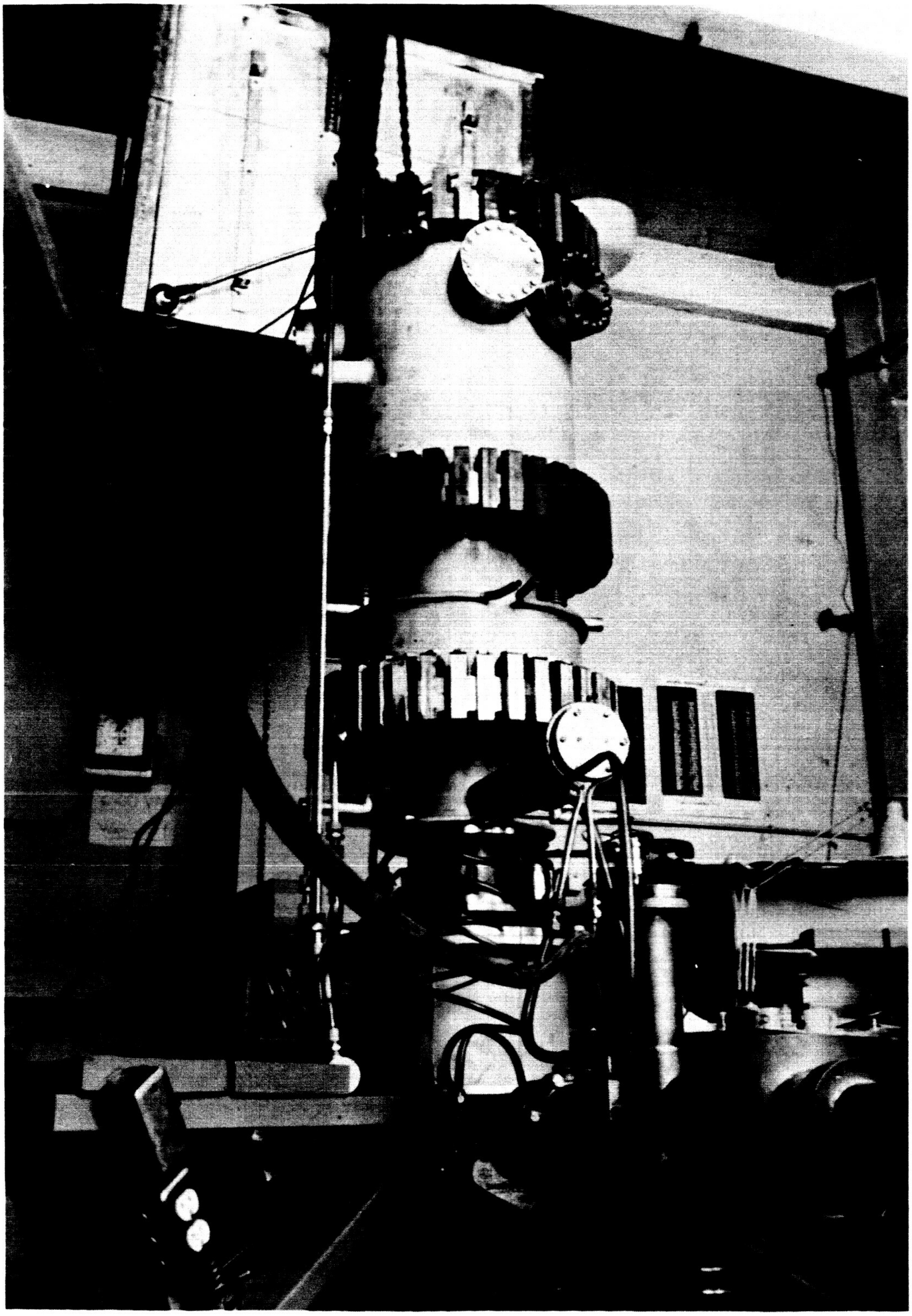

Figure 2. Ultra-High Vacuum System

gasket which is pinched between mating stainless steel flanges. For the larger seals (the 16" diameter chamber) a double pinch seal with a guard vacuum was employed. Figs. 3 and 4 show this.

In Fig. 3 we see a cross section of the single pinch seal used with the 2" and 4" OD tubing. The diameter of the sealing surface of the male flange (upper) exceeds the diameter of the female flange (lower) by 0.040", thus subjecting the copper gasket to enough pressure to deform it to the state shown in Fig. 3-b. The sealing pressure of approximately 6000-7000 lbs. per lineal inch of gasket is applied by means of 5/16" - 24 NF S. S. machine bolts which screw into a tapped split ring clamp. This eliminates both the danger of stripping the threads of a hole tapped in an expensive flange and the difficulties associated with using a wrench on nuts located in inaccessible places. Not shown are tapped jack screw holes in the male flange which proved essential to the separation of the flanges after a few bakeouts. When located in a vertical plane these seals present no difficulty in assembly as the gasket, being rigid, can sit quite comfortable in its recess if initially set at a slight angle.

Fig. 4 shows the double pinch technique with the fore vacuum pumpout located between the pinches. The land on the male flange is 0.040" wider than the groove in the female. Here symmetry requires that the sealing force be

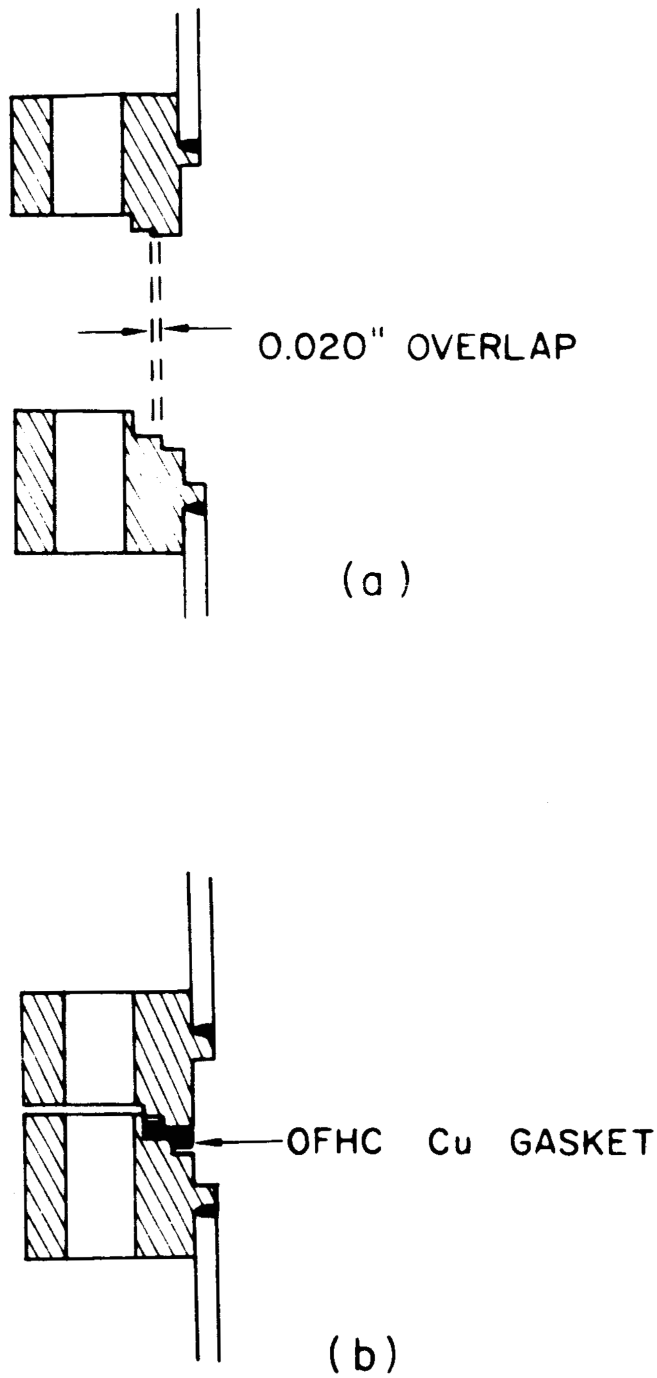

Figure 3. "Single Pinch" Seal

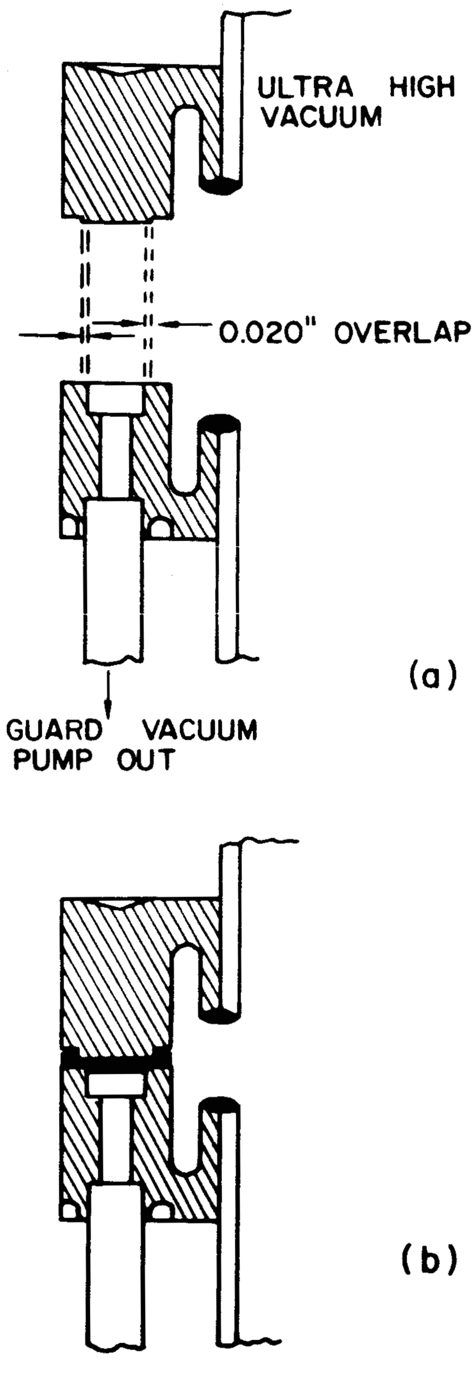

Figure 4. "Double Pinch" Seal

applied midway between the seals. Therefore, clamps machined from 1" thick 304 stainless steel bar stock and ½" - 13 NC machine bolts are utilized, as may be seen in Fig. 2. The clamps are spread about ½" apart and torqued to give an estimated sealing pressure of 5,000 lbs. per lineal inch of gasket.

The only exception to the above mentioned bakeable sealing pattern is a commercial gold "O"-ring seal used with the nude Bayard-Alpert ionization gauge mounted on the top of the chamber. For all seal variations "Silver Goop" (Crawford Fitting Co., Cleveland 10, Ohio) is used on the threads of the bolts to prevent galling. The non-bakeable seals are made with the conventional elastomer "O"-rings.

Three large diameter double pinch bakeable seals have been used: the first sealing the upper trap assembly to the lower trap assembly (the lower flange of the lower trap is water cooled), the second sealing the experimental chamber to the upper trap assembly, and the third sealing the lid to the experimental chamber. In addition, seven 4½" diameter seals and three 2½" diameter single pinch seals have been incorporated, amassing a total of 13½ lineal feet of double pinch seal and 10 lineal feet of single pinch seal.

<u>Baffles and Traps</u>. Immediately above the diffusion

pump inlet (see Fig. 1) is a chevron type water cooled copper baffle. The cooling water for the diffusion pump runs first through this optically dense baffle. The water cooled baffle is followed by a copper chevron type refrigerated baffle with a stainless steel oil creep barrier (Fig. 5). This baffle is maintained at about -40°C by a freon 22 refrigeration system. These baffles are run continuously and serve as the only oil trapping devices during system bakeout.

A typical liquid nitrogen trap is shown in Fig. 6. The liquid nitrogen reservoir, with a volume of 8½ liters, and its associated filling tubes are of stainless steel. The baffles themselves take the form of copper trays, capable of carrying a sorbant (Figs. 7 and 8), which are welded into a copper cylinder. This unit fits closely on the inside diameter of the liquid nitrogen reservoir, and is thus cooled primarily by radiation to the walls of the reservoir. S.S. radiation shields have been inserted between the reservoir and its warmer surroundings in order to maximize the life of the trap. The stainless steel oil creep barrier, welded at the top to the liquid nitrogen reservoir and at the bottom to the vacuum envelope, eliminates any warm path through the trap and simultaneously increases the mechanical rigidity of the trap, which would otherwise be supported only by the relatively light filling tubes. The pieces of flexible hose (0.030" thick

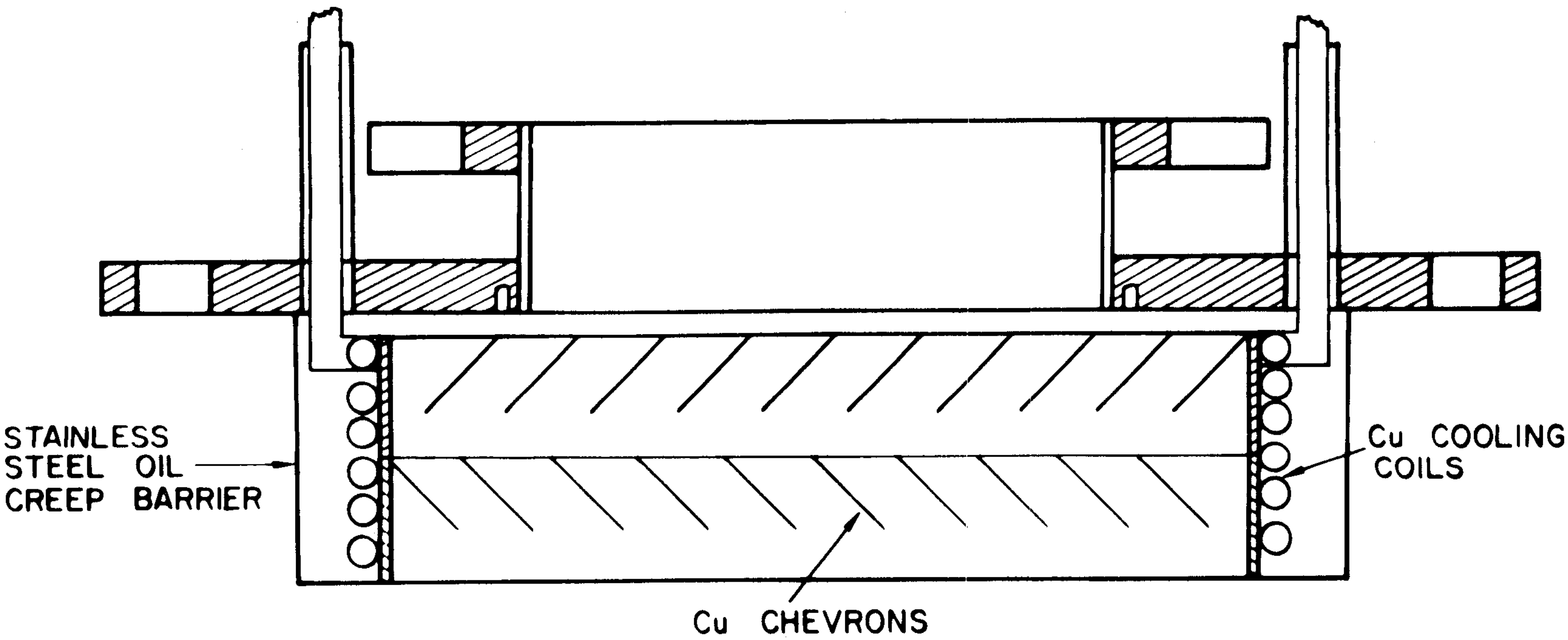

Figure 5. Freon Cooled Baffle

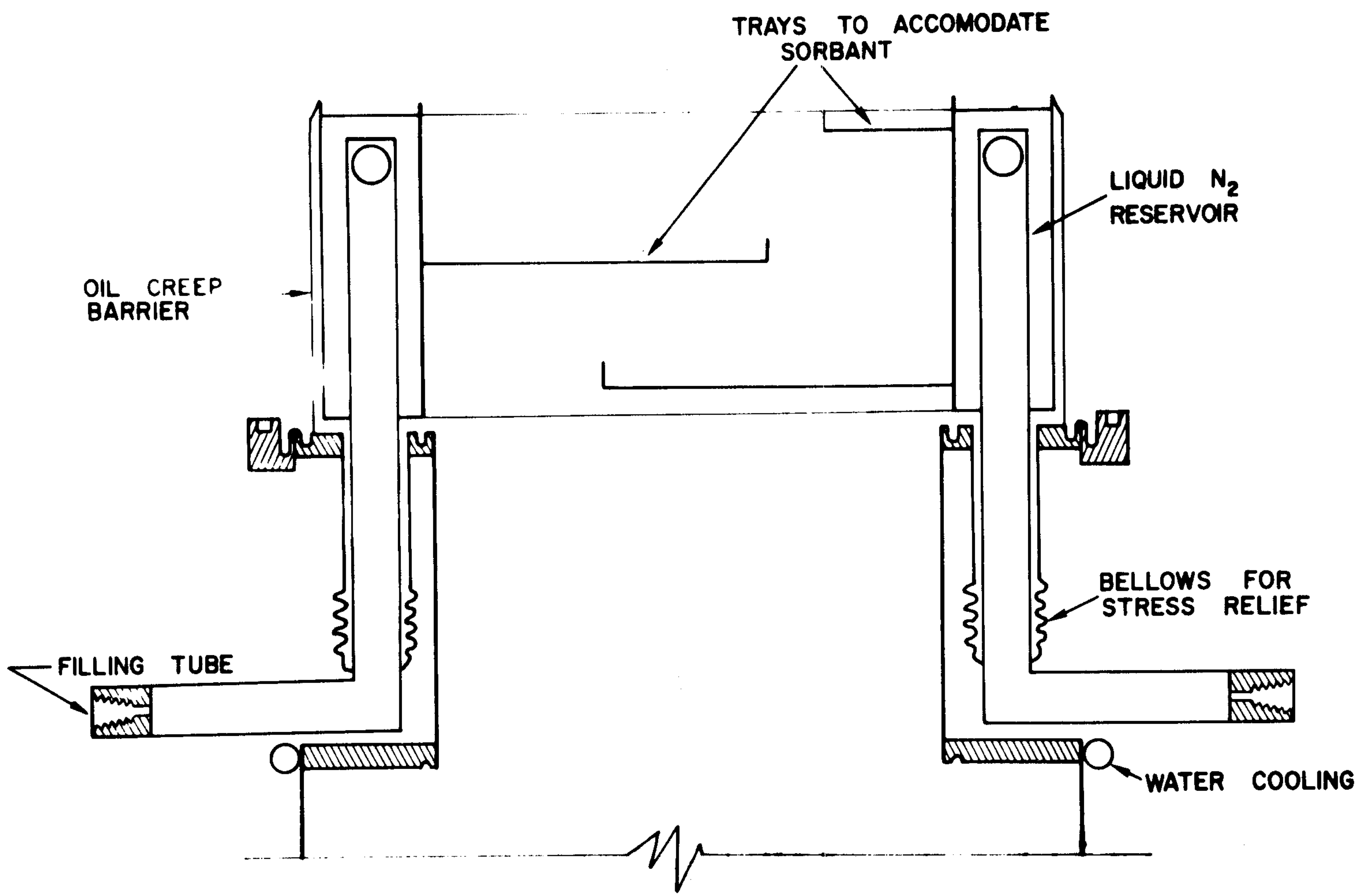

Figure 6. Liquid Nitrogen Trap

Figure 7. Zeolite Loaded Liquid Nitrogen Trap

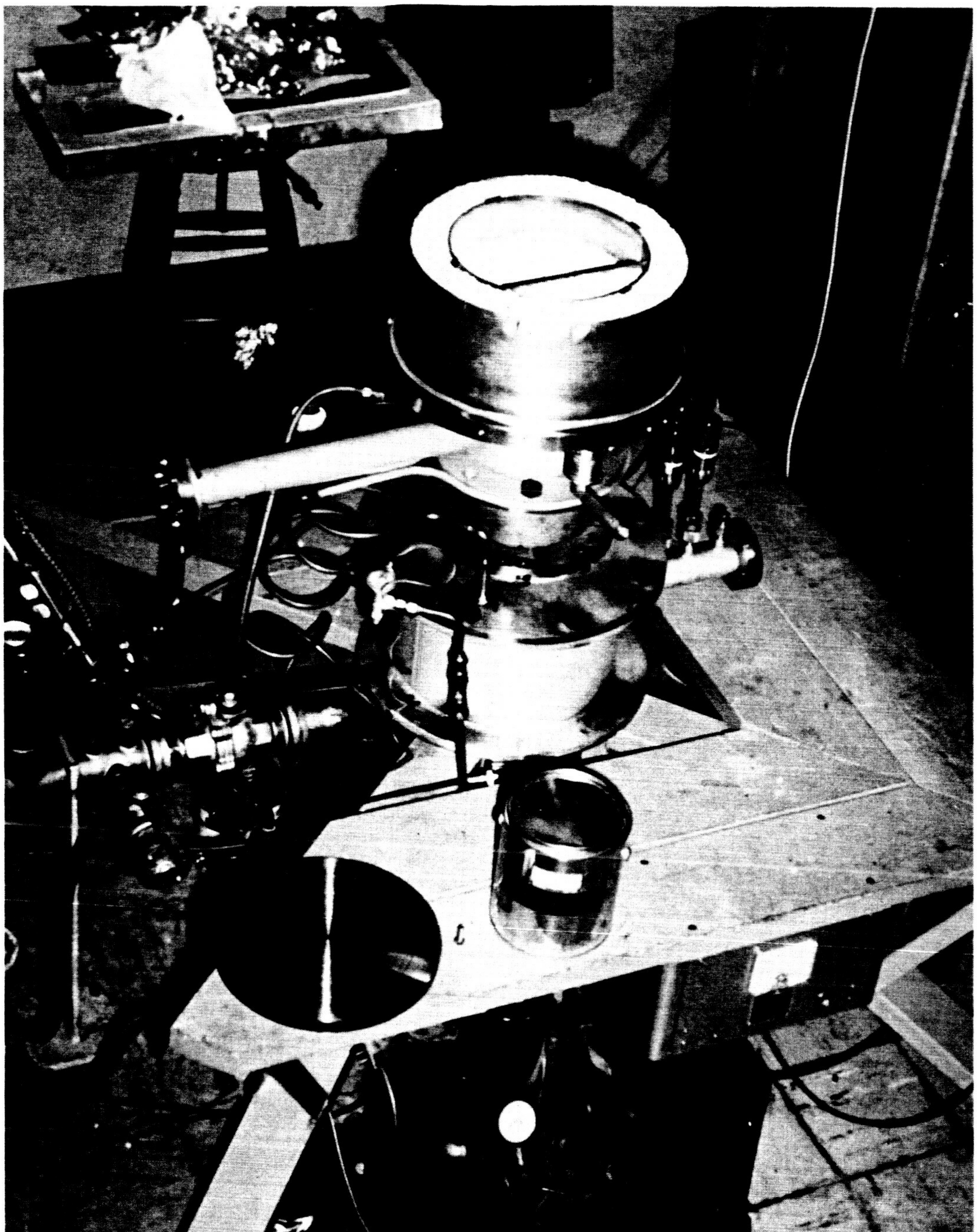

Figure 8. Partially Assembled Ultra-High Vacuum System

stainless) seen at the joint between the filling tubes and the vacuum envelope provide strain relief. Without this provision the filling tube to reservoir welds would have to endure stresses of the same order of magnitude as the tensile strength of stainless steel because of the temperature differential between the near room temperature oil creep barrier and the liquid nitrogen filled reservoir. The traps are thus a compromise between the strength and dependability of stainless steel and the high thermal conductivity of copper. In order to minimize the height of the vacuum system it was necessary that the traps be designed for bottom filling.

Ovens. The actual bakeout is accomplished by two "Thermobestos" (Johns-Manville) insulated ovens. (See Figs. 9, 10, 11.) The ovens are independently controlled by "Symplytrol" (Assembly Products, Inc., Chesterland, Ohio) proportioning temperature controllers. Each of the ovens is provided with 7 kW heating capacity by means of 8 incoloy sheathed, MgO insulated, nichrome spiral heated heating elements. Two of the elements in each oven are continuously controlled by the proportioning controllers. Gross temperature control is achieved by manually switching in additional heaters.

The lower oven (34" x 34" x 13") bakes the lower trap only, and is installed permanently in place with

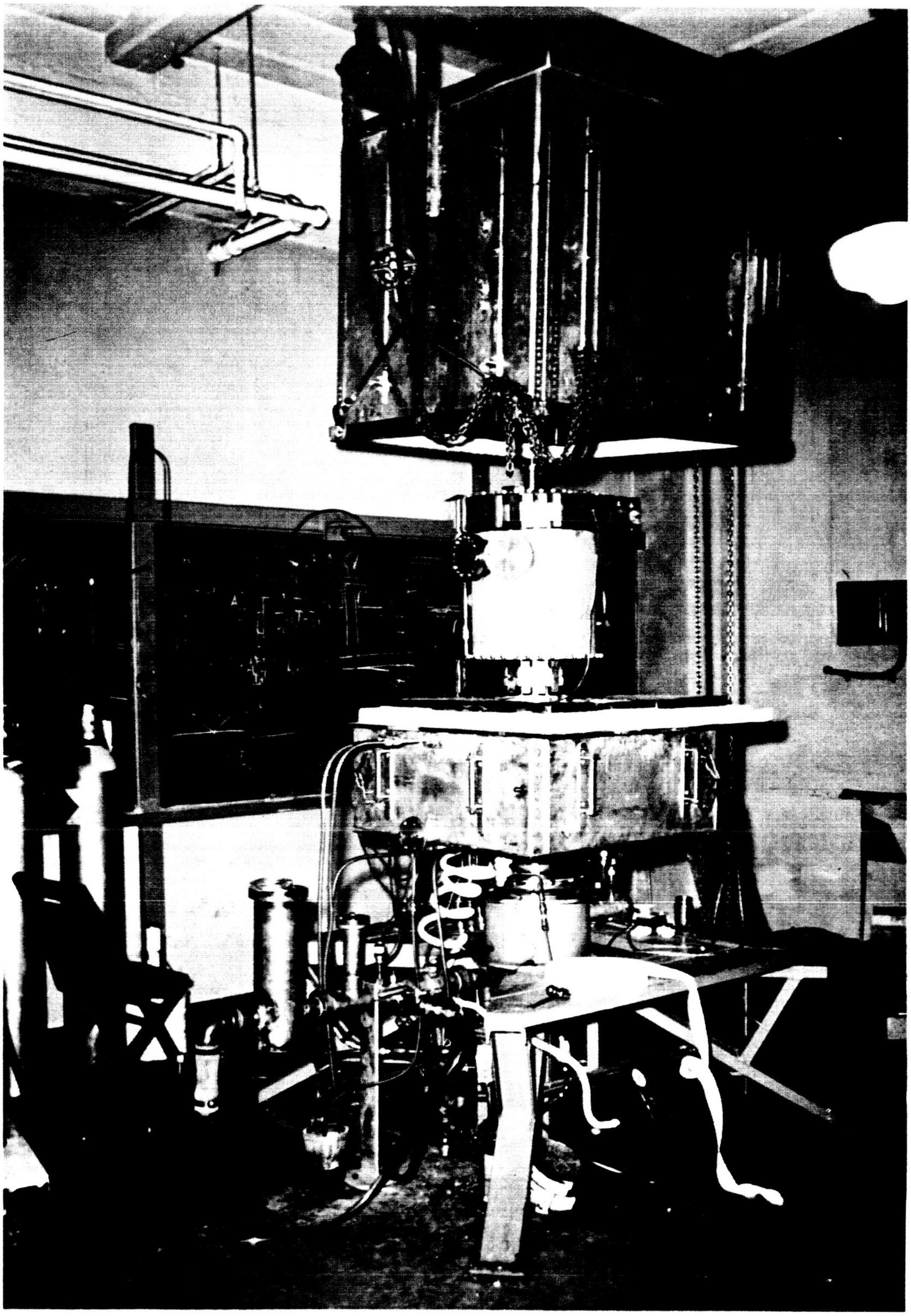

Figure 9. Assembled Ultra-High Vacuum System and Ovens

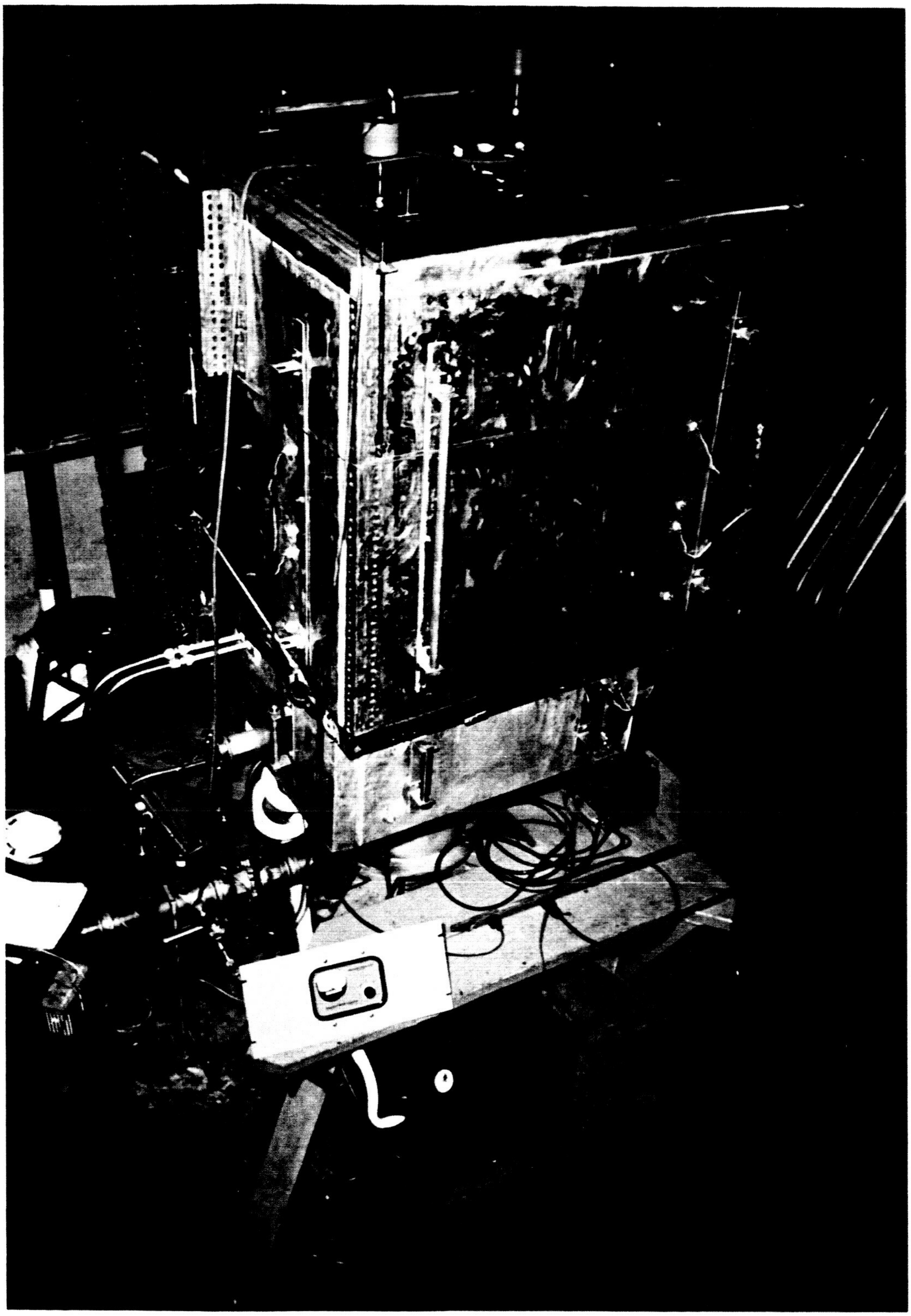

Figure 10. Ultra-High Vacuum System During Bakeout

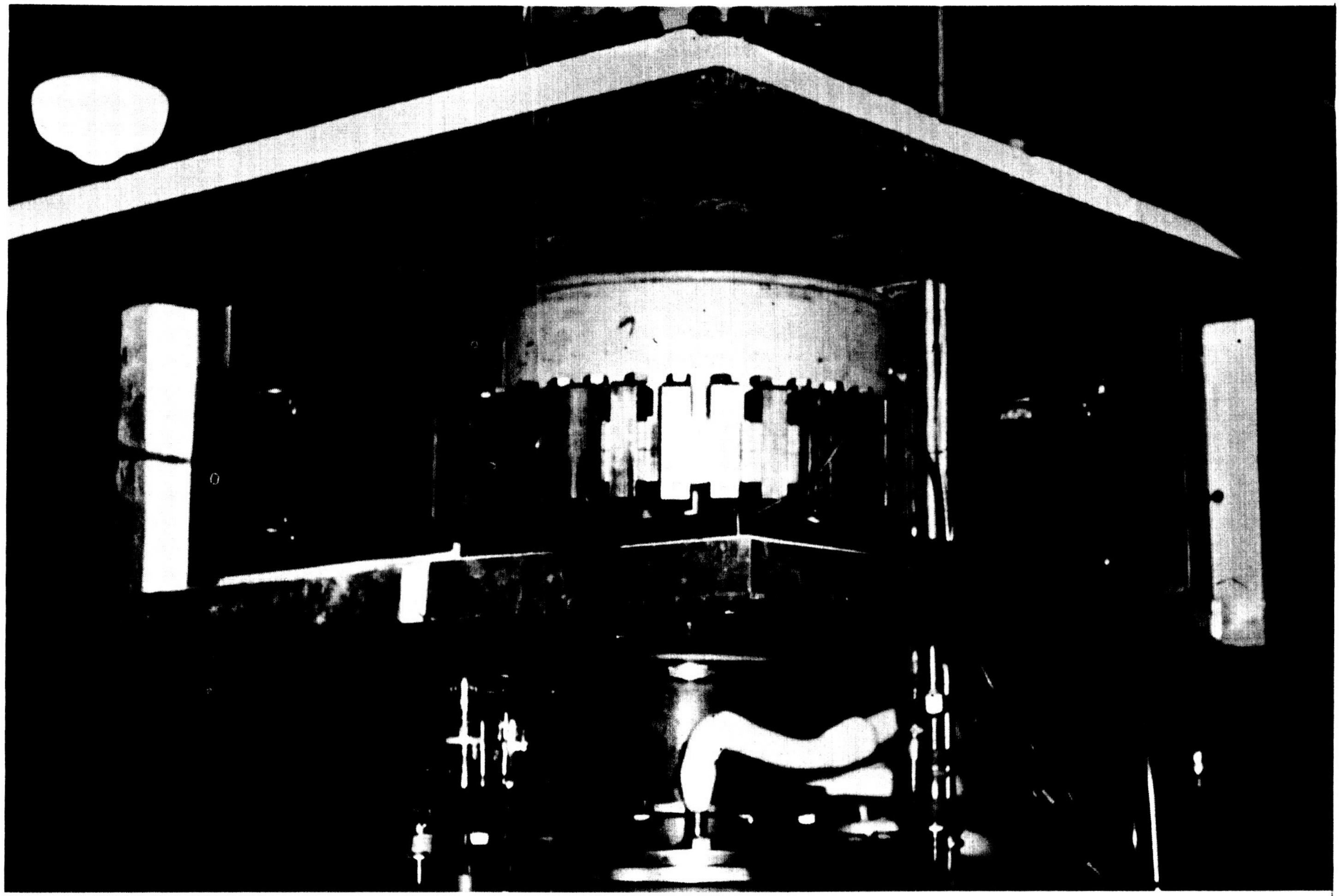

Figure 11. Inside Lower Oven

appropriate inlets for water cooling, liquid nitrogen, temperature measurement and control, etc. The upper oven (40" x 40" x 40"), which bakes the upper trap and ultra-high vacuum chamber itself, is supported from above by a pair of differential chain hoists, and can thus be lifted after bakeouts to allow access to the apparatus. Two fans have been installed in the roof of the upper oven to improve temperature uniformity. The shafts of the fans are lead through the four inch thick insulation to the motors outside by means of water cooled bearings. Ordinary machine oil serves adequately as lubrication.

Bakeout. The bakeout is performed in three steps. The system temperature is raised to 450°C at the rate of 25-30°C per hour. This temperature is maintained until the pressure, measured by a VG-1A ionization gauge which is installed in an unbaked section of the system, drops to the 10^{-7} Torr range. If the system is reasonably clean, this usually occurs after one or two days at maximum bakeout temperature. The lower trap is then cooled to room temperature and filled with liquid nitrogen. The bakeout of the upper trap and ultra-high vacuum chamber is continued for an additional 24 hours. The heaters of the upper oven are then switched off, and, after the temperature inside has dropped to about 200°C, the oven is lifted clear of the system. The Bayard-Alpert ionization gauges on the chamber are then degassed and the upper trap filled. The pressure at this time is in the 10^{-8} Torr range, after the upper trap is filled, the pressure drops to the 10^{-9} Torr range over a period of a few hours.

As mentioned earlier, the liquid nitrogen traps are loaded with a commercial sorbant (Consolidated Vacuum Corporation "Sorbant A"). After exposure to atmospheric pressure this sorbant is, of course, saturated. In addition, it has a strong affinity for water, and is, therefore, particularly inappropriate for a vacuum pump until after it has been activated. With a freshly loaded zeolite trap, the diffusion pump (unaided by liquid nitrogen trapping) cannot pump the system below the 10^{-4} range. Under this condition the foreline pressure is about 500 microns. A liquid nitrogen finger trap, which is visible in Fig. 9 to the right of the forepump inlet, has been installed in the foreline. When filled with liquid nitrogen, the foreline pressure immediately drops to about 15 microns. Inspection of this trap after a few hours pumping time reveals a 1/8" (approx.) thick coat of ice on the side of the trap facing the diffusion pump outlet.

The sorbant, which has been placed only in bakeable parts of the vacuum system, is activated by bakeout. During the initial hours of bakeout, the system pressure (with diffusion pump running) remains above a micron. It is possible, by continually increasing the temperature, to maintain this pressure (for maximum throughput) until the outgassing from the zeolite begins to decay. After the bakeout, a thick (about 3/4") mound of ice can be found on the liquid nitrogen trap in the foreline.

It is quite evident that the sorbant evolves great quantities of water during the activation process. Care must be taken that this water be kept out of the forepump, hence the liquid nitrogen trap in the foreline. Also, in order to maintain an efficient trap in the foreline, it should probably be cleaned several times during zeolite activation. This would be easily accomplished if a by-pass line around the foreline trap were to be added. As the system stands, it would be necessary to valve off the diffusion pump outlet during a time of very high throughput.

Bakeout and Liquid Nitrogen Control Circuitry. As has been mentioned, two commercially available temperature controllers control the oven temperatures. These same devices also form the nucleus of an automatic liquid nitrogen filling circuit. Under full bakeout conditions, they control the oven temperatures in a straight forward manner. In the "single bake" state, i.e., upper oven hot and lower trap cold, the relay contacts of the lower oven temperature controller TC-1 are switched into the liquid nitrogen control circuit and the controller input leads are switched from the controlling thermocouple in the lower oven to a thermocouple at a liquid nitrogen sensor which is mounted at the exhaust of the lower trap. A solenoid valve on the liquid nitrogen dewar opens upon a signal from a timer, the trap fills and overflows, and the temperature controller, sensing the presence of liquid nitrogen, closes the solenoid valve. When the bakeout is complete and the traps are cold, both of the temperature controllers serve to control the liquid nitrogen filling. The circuit is shown schematically in Fig. 12.

Attention should first be directed to S-2, the mode switch, which is shown in the "full bake" position. It is this switch which switches the temperature controllers progressively into the liquid nitrogen control circuit and switches the input thermocouples. Most of the poles of this switch go to the temperature controllers which are represented only by their barrier strip connections. Reading from left to right, "C", "NO", and "NC" are respectively the pole, the normally open contact and the normally closed contact of the controller relay. The circuit between "C" and "NC" is complete when the set point of the controller is above the temperature indicated by the controller thermocouple. The contact marked "110" and "C" are the 110 VAC power inputs, and "+" and "-" the thermocouple inputs. As the circuit is drawn, the controllers are connected to the 110 volt power line through S-2-D

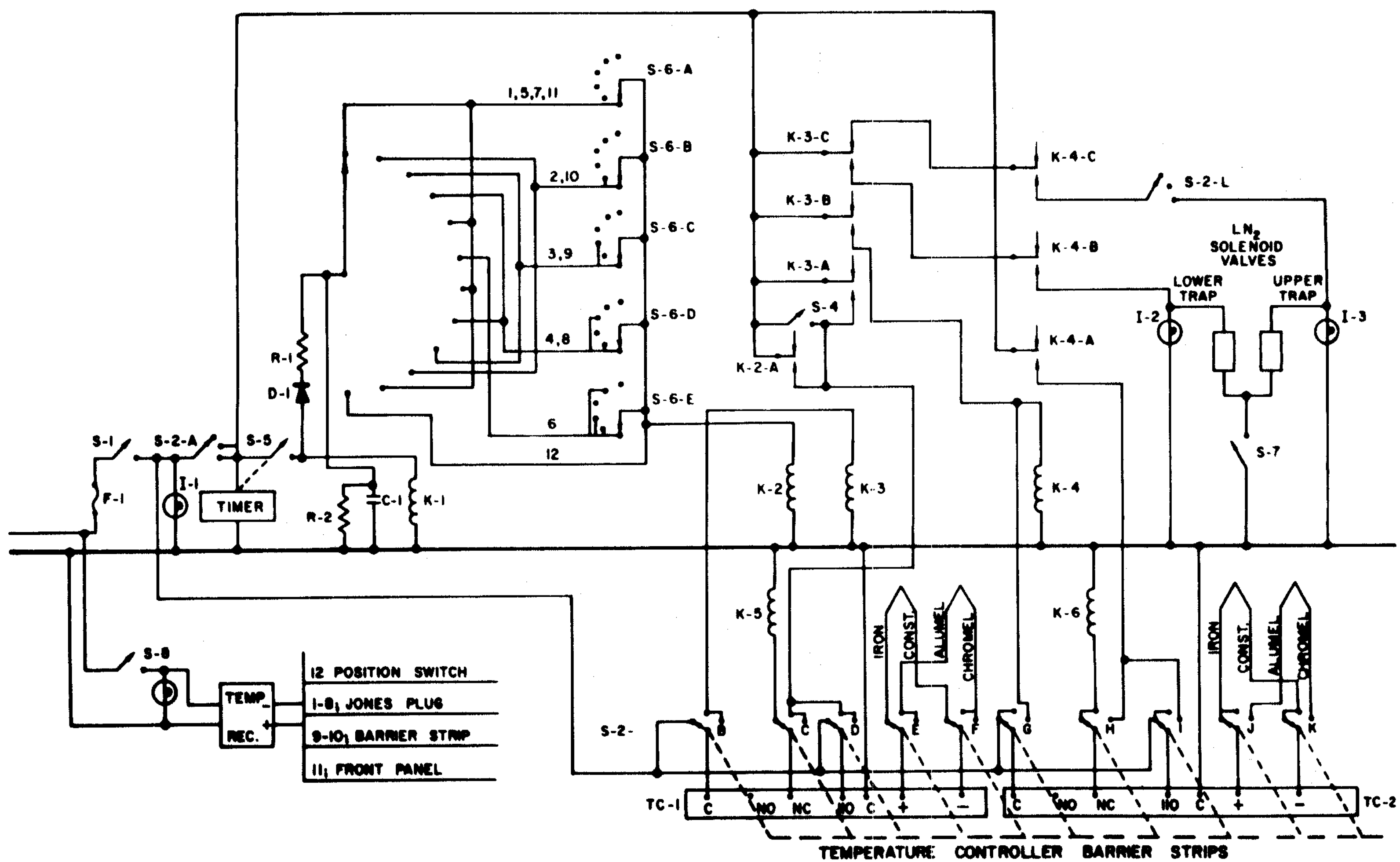

Figure 12. Liquid Nitrogen Filling Circuit

and S-2-I and to relays K-5 and K-6, which control heaters in the lower and upper ovens respectively, through S-2-C and S-2-H.

The action of the circuit with S-2 in its second position is as follows. Power is available to the timer which closes S-5 once each hour. When S-5 closes, power is available to the coil of the 12 position stepping relay, K-1. Also, D.C. voltage is applied to the pole of K-1, but delayed by the charging time of C-1 through R-1. This short delay is necessary because the D.C. voltage must not be applied before K-2 has had time to complete its stepping action. Thus, once every hour D.C. voltage is available at a different contact of K-1. These contacts are wired to S-6 ("Imterval" switch) so that when S-6 is in its first position (as shown), it will transfer this voltage to the coil of K-2. If S-6 is in its second position, it will transfer the voltage only every other hour. In the third position, S-6 transfers every three hours, and similarly 4, 6, and 12 hours for positions 4, 5, and 6. Thus the interval between trap fillings is variable from 1 to 12 hours.

Whenever S-4 (SPST mom. "Manual Fill" switch) is closed or when K-2 is energized, power is made available, through S-2-D to TC-1 and, through S-2-C, to the "NC" Contact of the relay of this controller. The output of the chromel/alumel thermocouple (which is connected so that its output is positive for temperatures below room temperature) at the trap exhaust will be low. Therefore, the controller will see a temperature lower than its set point (which should be about 100°C in this application) and will complete the circuit between "C" and "NC". Power will then be available to K-3 through the series circuit consisting of S-2-C, the controller relay, and S-2-B. Relay K-3 holds itself by means of K-3-A and provides power to K-4 by means of K-3-B. With both K-3 and K-4 energized, power is available through K-3-C and K-4-B to the solenoid valve for the lower trap. When the trap is filled, the chromel/alumel thermocouple is chilled and the resulting signal to TC-1 causes it to open the C-NC circuit, which in turn releases K-3 and K-4, so that the solenoid valve closes. In this mode, the upper oven controller TC-2 remains in the same state as in the full bake mode.

In the "double trap" mode, the operation of the circuit is similar except that now, K-4, once energized, can hold itself independently of K-3, through the circuit consisting of K-4-A, S-2-H, the upper oven controller relay, and S-2-G. Therefore, when the lower trap is full, the signal from the chromel/alumel thermocouple at the lower trap, causes only K-3 to be released. In this state, the circuit through K-3-C and K-4-C is complete to the upper trap solenoid valve. When this trap is full, TC-2 releases K-4 and the cycle is complete. S-2-L merely insures that no voltage will be applied to the upper trap solenoid valve unless the system is being operated in the "double trap" mode.

Switch S-7 is actuated by the pressure in the liquid nitrogen dewar. It opens if the pressure drops below a preset level, i.e., if the dewar is emptied. If this should occur while, say, the lower trap is filling, the opening of S-7 puts the coils of the two solenoids in series with pilot light I-3. This greatly reduces the power dissipated in the solenoids and prevents them from over heating. The circuit will remain in this state until given human attention since TC-1 would never receive a signal indicating a full trap. The fact that the dewar is empty is immediately clear to an operator since <u>both</u> pilot lights, I-2 and I-3, will be on, the brighter of which indicates which trap was filling when the dewar went dry.

The liquid nitrogen overflow sensor, Fig. 13, is designed so that the thermocouple is shielded from the blast of cold but gaseous nitrogen which precedes the filling of the trap. Most of this blast is directed out through the large holes in the walls of the (plexiglass) cylinder. When the liquid nitrogen reaches the sensor, it is able to flow through the small axial hole near the center and thus chill the thermocouple mounted below.

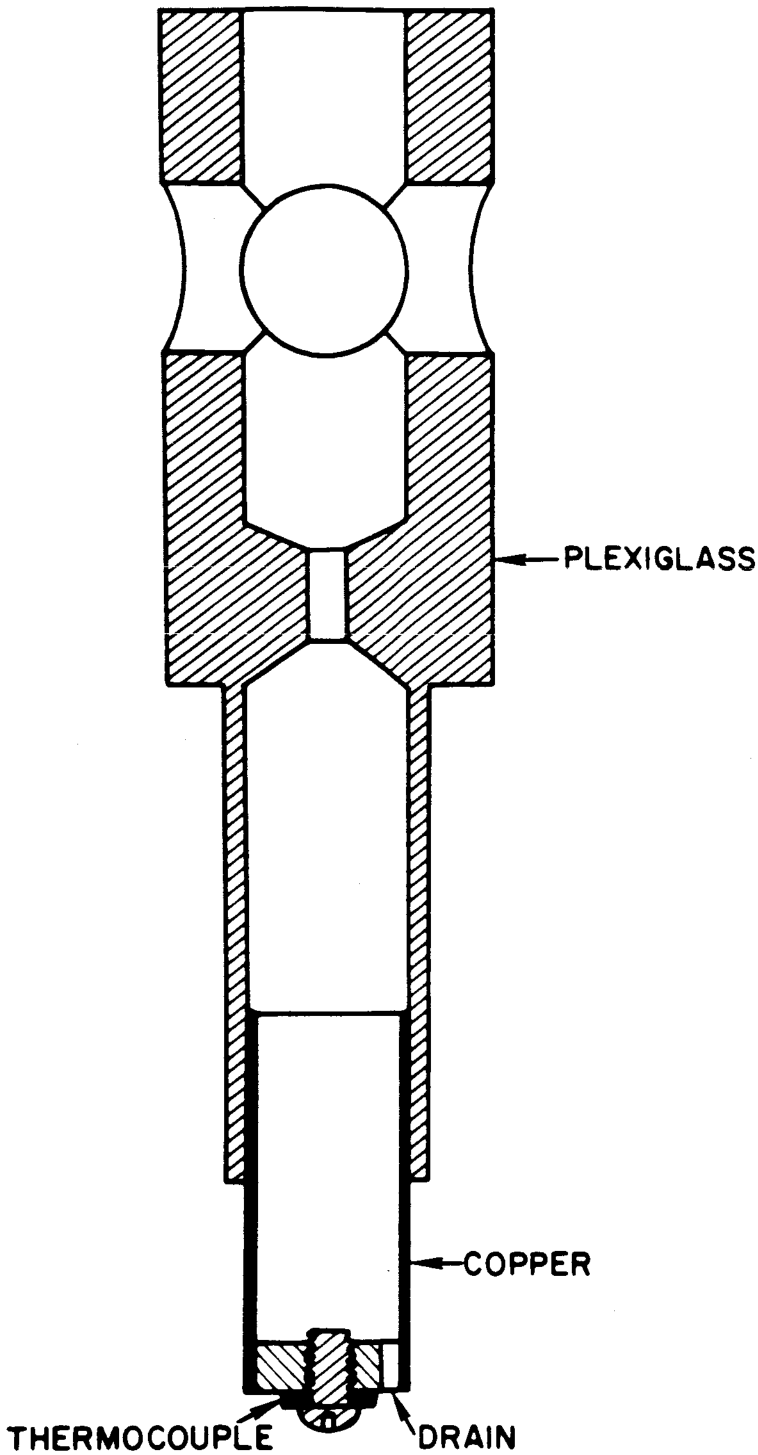

Figure 13. Liquid Nitrogen Overflow Sensor

<u>Interlock Circuitry</u>. It was found that a momentary pressure burst, of even a few Torr is sufficient to permanently degrade an ultra-high vacuum to the 10^{-8} Torr range. Also, due to the limited availability of electrical power it was necessary to wire the system in a manner that was less than optimum. For example, the diffusion pump is connected to one side of a 220 VAC line, while the forepump and refrigeration compressor receive power from the opposite of the 220 line and are fused independently. Therefore, if a momentary power failure were to occur (in the absence of the precautions which have been taken), the combined starting loads of the forepump and refrigerator could blow the main fuse on that phase of the 220 VAC line. Thus the diffusion pump, still receiving power, would continue to operate while the vacuum system was slowly vented through the now ineffective forepump.

For these reasons, fairly extensive preventive measures have been taken in order to minimize the probability of accidental venting of the system. The interlock circuitry, shown in Fig. 14, provides the following features.

In the event of a power failure on the forepump side of the line, power is immediately removed from the diffusion pump and a pneumatic valve, which is installed in the foreline, closes. If the duration of the power

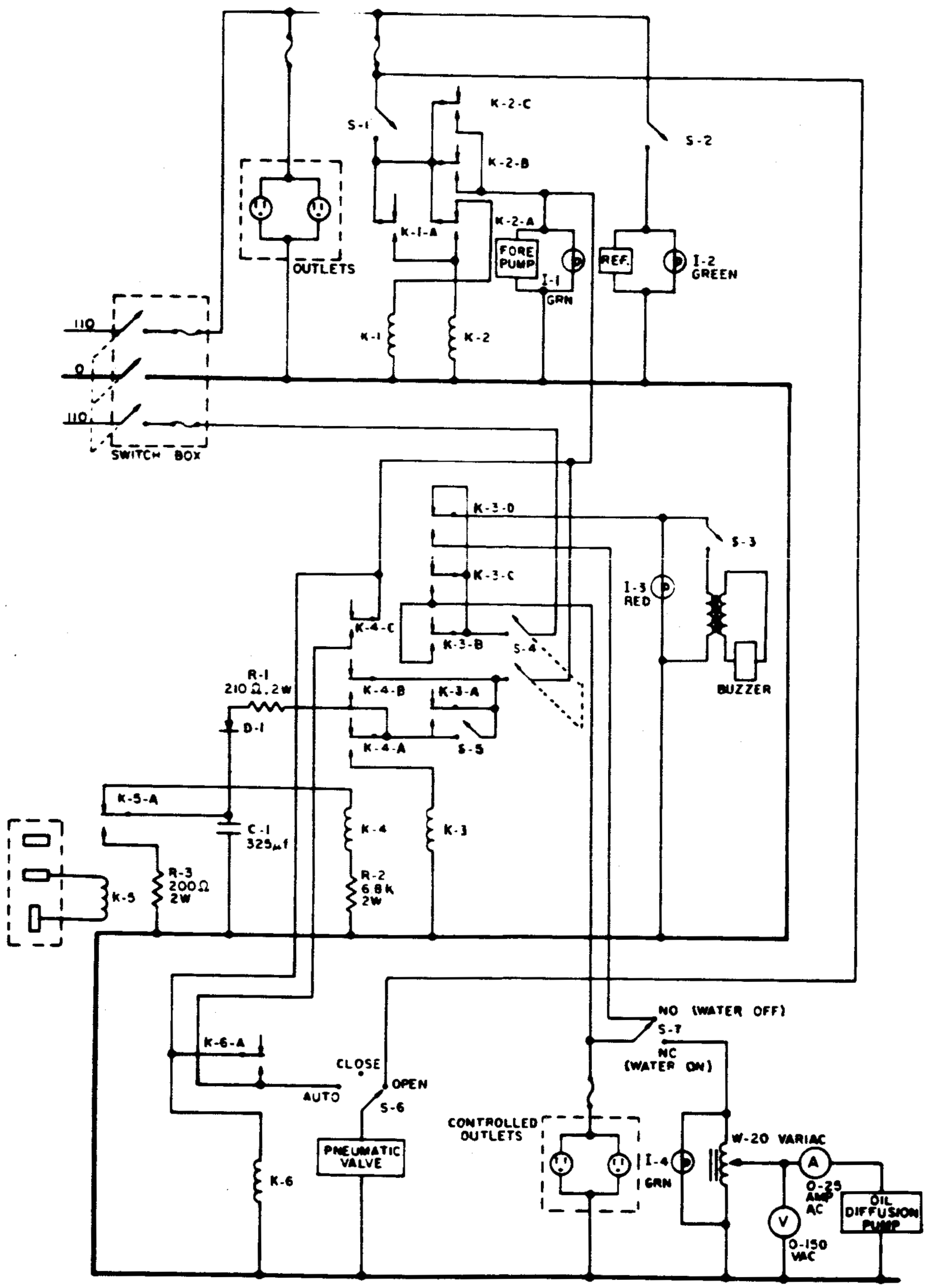

Figure 14. Interlock Circuitry

failure is less than five seconds, restoration of the power will put the vacuum system back into full operation, but in a definite order. When power returns, it is made available to the refrigerator but not the forepump, which is delayed by two seconds. The starting current of the refrigerator is quite high if the pressure differential of the refrigerant across the compressor has not had time to equalize; therefore, the refrigerator is given sole access to the power lines during this two second period. In this time it will either succeed in restarting or it will activate its own magnetic multibreaker and remain out of operation until given human attention. After the two second delay, the forepump and diffusion pump start again and the pneumatic valve opens. The incorporation of the five second delay avoids the annoyances involved with system shut-downs triggered by short power failures and line fluctuations.

In the event of a longer power failure, say an hour, the diffusion pump does not restart. However, the refrigerator and forepump (which, by itself, can maintain a system pressure in the low 10^{-5} Torr range) follow the two second delay cycle described above, but with the following difference. The opening of the pneumatic valve is delayed another thirty seconds to permit the forepump to evacuate the section of foreline between the forepump and the pneumatic valve, a small volume that could easily leak up to atmospheric pressure during an extended power failure. In addition to the above mentioned protection, the diffusion pump is shut off if the cooling water for it fails, or if the pressure in the vacuum system rises above a preset value.

Let us now examine the circuit in detail. When S-1 is closed, the two second time delay relay K-1 receives power through K-2-A. After two seconds, the contacts of K-1 close and thus energize K-2, which now holds itself by means of K-2-A. When K-2 is energized, power is removed from K-1 and is provided at one of the poles of DPST switch S-4.

When S-4 is closed, power from the forepump side of the line is available at S-5 and K-3-A. Also power from the diffusion pump side of the line is available at K-3-B, K-3-C, and K-3-D. Since K-3 is, at this time, de-energized, the circuit at K-3-D to the alarm assembly (buzzer and red light) is closed, therefore the red pilot light and (if S-3 is closed) the buzzer will be energized. If now S-5 (SPST Mom.) is closed, C-1 is charged through R-1 and D-1. When the voltage across C-1 is sufficiently high, K-4 is energized through the contacts of K-5, this in turn energizes K-3 through K-4-A. K-3 now holds itself through its own contacts, K-3-A. When K-3 is energized, power from the diffusion pump side of the line is provided to the pole of S-7 by means of the parallel connected

K-3-B and K-3-C contacts of K-3. SPDT switch S-7 is a microswitch on a commercially available flow rate interlock ("Shur-flo", Hays Manufacturing Co., Erie, Penn.) in the diffusion pump water cooling line. If the flow rate is sufficient, S-7 is in its normally closed position and power is available to the diffusion pump. If the flow rate is low, S-7 completes the circuit to the alarm system through K-3-D of (the now energized) K-3. In the event of a pressure rise in the vacuum system, 6.3 VAC is supplied to K-5, which immediately disconnects K-4 and discharges C-1 through R-3. The release of K-4 de-energizes K-3, thus removing power from the diffusion pump.

In the event of a power failure in the forepump side of the line, all of the relays are released with the exception of K-4, which is maintained for about 7 seconds by the discharging capacitor, C-1 (R-2 serves only to maximize the delay time). If power returns within seven seconds (N.B., the two second delay at the forepump reduces the effective receovery time of the circuit to five seconds) K-3 is energized through the series circuit comprised of S-4, K-4-B, and K-4-A, thus starting the diffusion pump. If power returns after the release of K-4, the diffusion pump will not restart.

Let us now turn our attention to K-6, S-6, and the pneumatic valve. The valve is "wired" electrically and pneumatically so that it closes when power is removed from it. The last two positions of S-6 provide manual control of the valve. When S-6 is in the first (auto) position, the pneumatic valve cannot be energized unless K-2 is energized. Also there is a direct connection to the valve only if K-4 is energized. Thus, for a power failure lasting less than 5 (= 7 - 2) seconds, the valve opens immediately upon reapplication of a.c. power to the circuit. If, however, K-4 has dropped out, the return of power closes K-2, after the two second delay introduced by K-1, but power is available only to the coil of K-6 (30 second time delay relay). Therefore, the valve does not open until after an additional delay of 30 seconds. Note that the actual time difference between restart of the forepump and the opening of the pneumatic valve, in the case of a power failure sufficiently long to prevent the restart of the diffusion pump, is a function of the duration of the failure. This occurs because K-6, being a thermal delay, can only realize its rated delay time when it starts cold. As K-6 is installed, it runs continuously during normal operation, and is always hot. Therefore, after a failure of only a few minutes, the delay may be only 10 seconds instead of the rated 30 seconds. This does not in any way negate its effectiveness because the sole function of K-6 is to insure that the pneumatic valve does not open before the section of foreline between valve and forepump has been reevacuated. Thus the relay actually proportions its action to the duration of the power failure.

Experimental Arrangement. A top view of the experimental chamber (right-hand circle), which has a useful volume of about 50 liters, is shown schematically in Fig. 15. The ports shown with detectors and the entrance port have been fitted with sapphire windows (Ceramaseal, using Linde UV grade sapphire) thus allowing for transmission and reflectivity measurements both near normal and near grazing incidence from 50,000Å down to 1500Å.[20]

The remaining port mounts an evaporation source. Five flanges have also been mounted on the lid of the vacuum chamber. Two provide miscellaneous electrical feed throughs, two mechanical rotary feed throughs (Cooke Vacuum Products), and one mounts a nude Bayard-Alpert type ionization gauge.

The existing ultra-high vacuum system is shown in a proposed application in conjunction with a normal incidence (17.5°) vacuum UV monochromator (left-hand circle). The monochromator will supply monochromatic radiation in the range between 1500Å (sapphire cutoff) and 2000Å or longer. Reflectivities in this wavelength range are to be measured under ultra-high vacuum conditions at two angles of incidence θ_1 (17.5°) and θ_2 (55°). The data thus attained being sufficient to determine the optical constants of the sample. It is proposed that the first

[20] Linde Industrial Crystals Bulletin, 5/15/62, F-917-C.

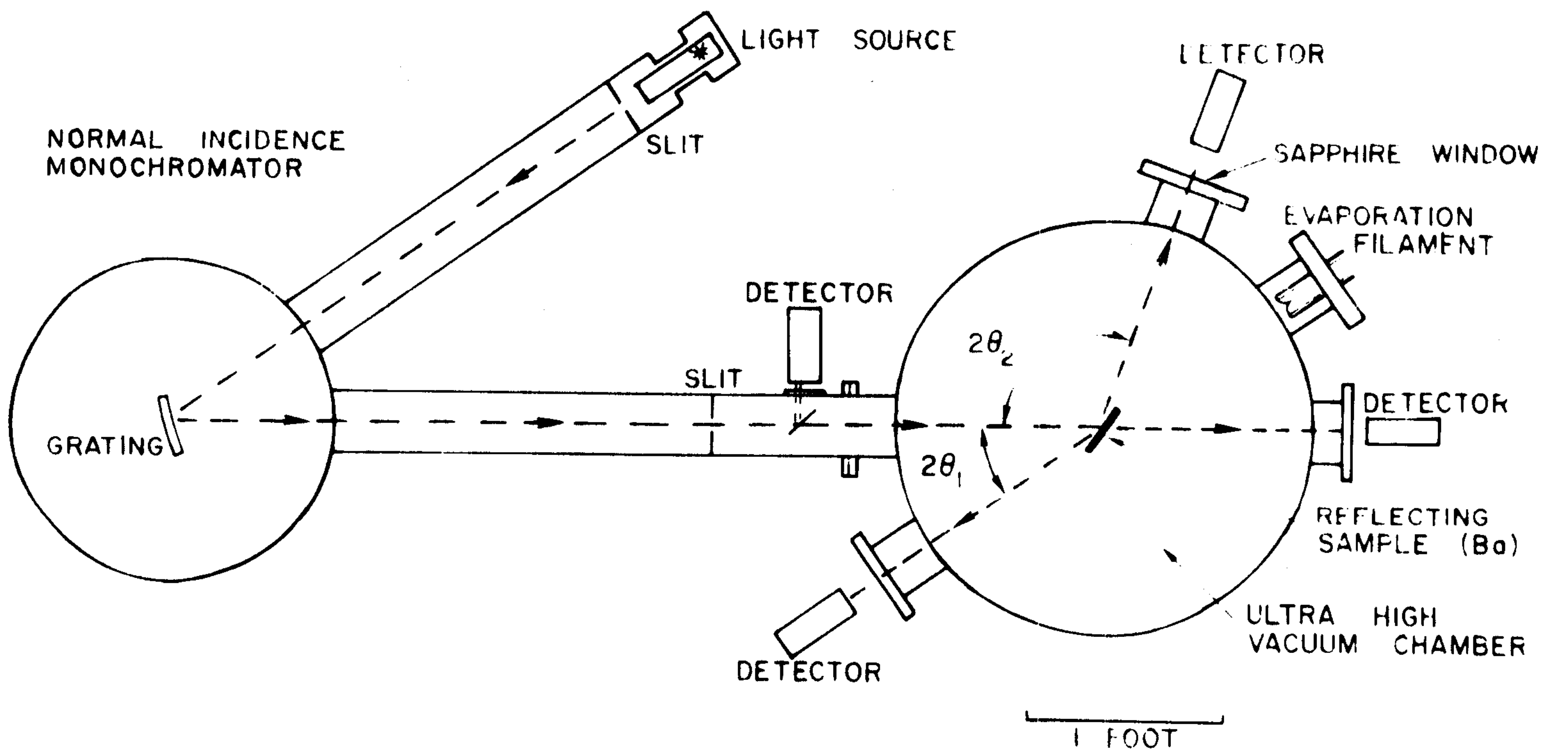

Figure 15. Experimental Arrangement for Measuring Reflectivities under Ultra-High Vacuum Conditions

studies be made with Ba, because of the availability here of radiation between the quartz and sapphire cutoffs and because the optical properties of barium should show interesting behaviour--due to its plasma loss at 6.5 eV[21] in this wavelength range. A sapphire window is provided directly opposite the entrance port for transmission measurements and determination of the incident intensity.

It should be mentioned that the ultimate goal is to arrive at an experimental arrangement similar to that shown in Fig. 2 in which both the monochromator and the experimental chamber are maintained at ultra-high vacuum. (That is, the pressure in the monochromator is sufficiently low that it is possible to maintain the experimental chamber at ultra-high vacuum without the necessity of an intermediate window.) Then it will be possible to make measurements throughout the entire range of the vacuum UV. It is our intention that the θ_2 and the transmission ports become the entrance and exit arms of a Seya monochromator and that the present experimental chamber become the grating chamber of the final ultra-high vacuum apparatus. In this case, the short wavelength limit would be determined only by the light source and the inherent limitations of the Seya mounting.

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[21] J. L. Robins and P. E. Best, Proc. Phys. Soc. 79, Part 1, 110 (1962).

<u>Transition</u> <u>Radiation</u>. In addition to the above described approach to the optical properties of metals, a ½-meter normal incidence vacuum UV spectrograph is being used to investigate the transition/plasmon radiation phenomenon in aluminum. The apparatus is shown schematically in Fig. 16. Pure Al films are evaporated in situ at a pressure of about 2×10^{-5} Torr. The films, which are evaporated onto a stainless steel target, and bombarded with 25 keV electrons, are 600 - 1000Å thick, and hence much thicker than the optimum value (about 60Å in this case) predicted by Ferrell.[7] Therefore, the experiment is really an investigation of transition radiation in what is effectively bulk aluminum.[22,23]

Up to the present, a cathode ray tube type electron gun has been used as an electron source. Since this gun uses a weakly heated oxide cathode, the danger of exposure of the grating to stray light is greatly minimized. In addition, the gun is inexpensive and reasonably convenient to install. Unfortunately, the maximum target current that has been obtained so far has been about three microamps. This results in enough radiation to give a fairly dense central image (possibly detectable with a sensitive photomultiplier) but not enough intensity to give any

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[22] R. A. Ferrell and E. A. Stern, J. Quant. Spectros. Radiat. Transfer <u>2</u>, 679 (1962).

[23] R. H. Ritchie and H. B. Eldridge, Phys. Rev. <u>126</u>. 1935 (1962).

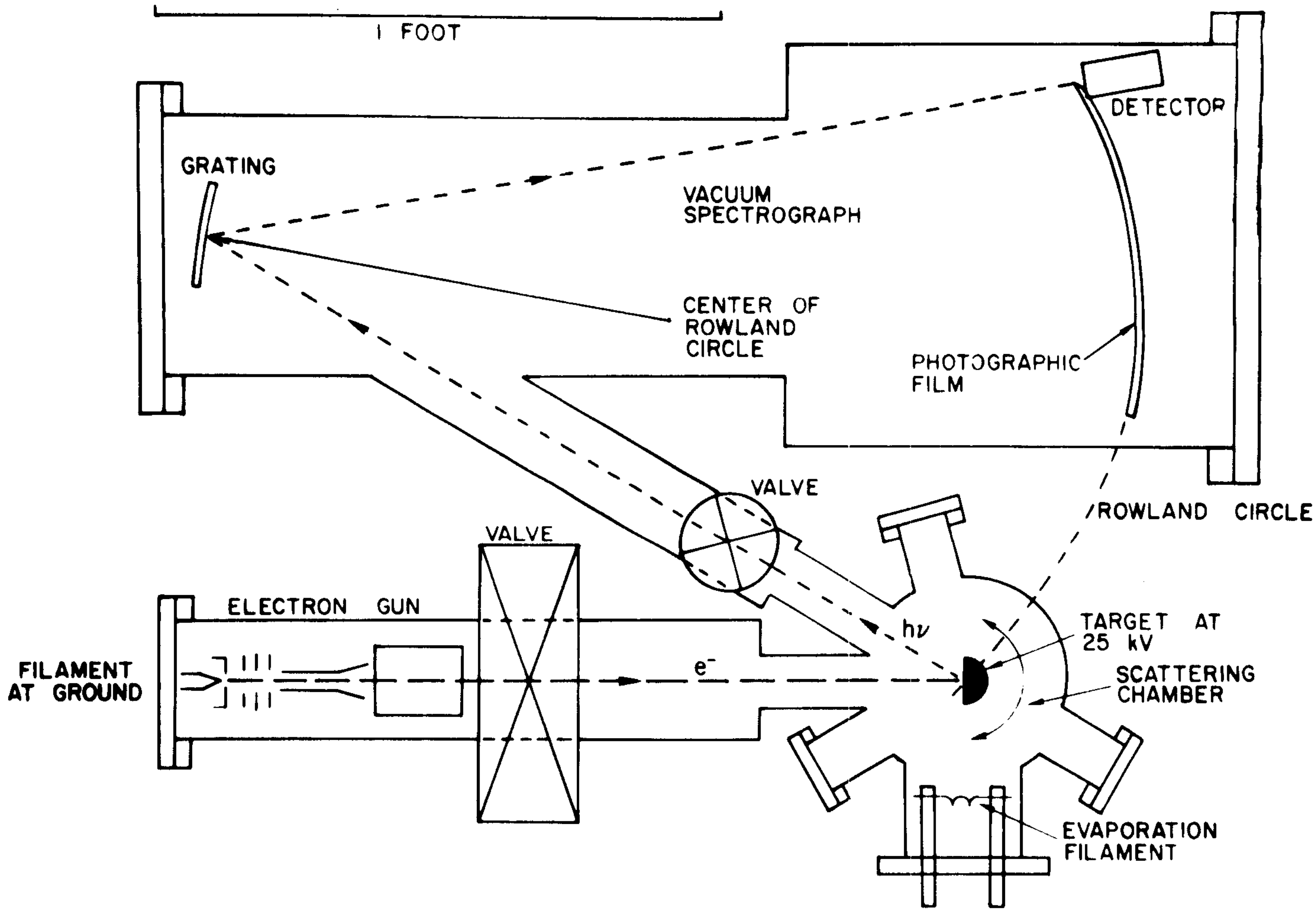

Figure 16. Experimental Arrangement for Investigating Transition Radiation from Al

indication of the dipersed continuum. It will probably be necessary to go to a more specialized electron gun in order to observe any dispersed intensity. Since the target rod is maintained at +25kV the problem of exposure due to scattered electrons is eliminated. In this interest a 300 line/mm grating has been ordered to replace the 1200 line/mm grating that is now in the spectrograph.

Initial filter measurements have been encouraging. Most of the radiation is definitely at wavelengths shorter than the glass cutoff. Experiments with quartz and LiF have been less positive. Although more data need to be taken, it appears as if there is radiation at wavelengths shorter than 2000Å. Information about wavelengths shorter than LiF, of course, is that much more indefinite. The expected spectrum is a continuum with a peak at about 800Å which tails off quickly to shorter wavelengths and slowly to longer wavelengths (extending almost to the visible). A further rather encouraging piece of datum is the following. The radiation in a direction normal to the film should be zero (for normally incident electrons). One exposure has been made for normal radiation. This showed less intensity than a similar exposure for radiation emitted in a direction 30° from the normal.* It should be pointed

*One would expect to observe some radiation in the forward direction because of the finite solid angle subtended by the grating and extraneous sources of radiation such as bremsstrahlung.

out that the predicted radiation null in the normal direction is for normally incident electrons and is correspondingly difficult to observe. For this experiment, when the radiation is observed normally, the electrons are incident at 30° and vice versa.

A further filter measurement that should be quite informative is planned. Indium has a transmission window between 11.1 and 16.8 eV.[24] An 800Å thick film showed a 17% transmission characteristic at 16 eV. This transmission window, which is approximately centered on the expected 15 eV plasma energy in aluminum, offers an almost ideal bandpass filter.

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[24]G. L. Weissler, W. C. Walker, and O. P. Rustgi, J. Opt. Soc. Am. 49, 471 (1959).

CHAPTER III

SUMMARY

We are attacking the optical constants of metals problem in two ways in this laboratory. We will attempt to measure reflectivities of clean metal surfaces under ultra-high vacuum conditions. Measurement of the reflectivity at two angles (with known polarization) permits calculation of the two optical constants n and k by means of the Kramers-Kronig dispersion relations. A second application of the ultra-high vacuum system is the measurement of the optical constants of the reactive alkali metals which are difficult (if not impossible) to handle under ordinary high vacuum conditions.

We are also looking for transition radiation in aluminum. Since the plasma frequency of Al (and many other metals) is in the vacuum UV spectral range, the use of an appropriate spectrograph is essential. We are well equipped with vacuum UV optical equipment, and the application to the transition radiation experiment is natural, considering the success of Steinmann[8] with silver (3300Å). It is necessary here to study transition radiation in thick aluminum films rather than the plasmon radiation predicted by Ferrell for thin films, because of the difficulties associated with maintaining an Al film (unsupported) of 60Å thickness.

It should be possible, sometime, to look for transition/plasmon radiation in the alkali metals using the ultra-high vacuum system. These two techniques (reflectivities and plasma frequency studies) give us two independent approaches to optical constants. It would be ideal, of course, to make both types of measurement on the same sample. Perhaps this will eventually be possible with the ultra-high vacuum system.